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Titrimetric analysis of total mercury ions including mercury(I) ions

Xin-Gui Li, Qiang Dou, Mei-Rong Huang

Key Laboratory of Advanced Civil Engineering Materials, Institute of Materials Chemistry, College of Materials Science and Engineering, Tongji University, Shanghai, China

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Abstract A systematic analytical method is proposed and applied to directly determine the total concentration of Hg(II) and Hg(I) ions in water. Experimental results demonstrate that this method provides a low detection limit of 0.05 m*M* and small relative error within 1.5% in an ion concentration range of 0.2–50 m*M*. The technique is especially applicable for sample solutions that the traditional titration method like *Volhard* and *EDTA* complexation titrimetry could not analyze directly. This method could be employed to analyze solutions in any ratio of Hg(II) and Hg(I) ions including pure Hg(II) or pure Hg(I) ions, exhibiting several advantages, such as simple operation, good reproducibility, and low cost.

Keywords Mercury; Mercurous ion; Total mercury concentration; Analysis of total mercury ion; Titrimetry.

Introduction

Heavy-metal ions including mercurous ions are among the most feared contaminants in water sources that pose severe public health problems nowadays [1, 2]. It is known that the mercurous ions released into the environment influence ecological life due to their accumulation and high toxicity in living organisms. The primary quantitative methods of mercury determination generally include instrumental [1–4], gravimetric, and volumetric analyses. Atomic spectral [5–8], chromatographic [3, 9], adsorptiometric [10, 11], electrochemical [12–15], and neutron activation analyses [16] are the most important instrumental methods. Among them, cold-vapor atomic absorption spectrometry [5, 6, 17, 18] and atomic fluorescence spectroscopy [19–21] are usually combined with chromatography in mercury analysis [22]. These coupled techniques, which combine high sensitivity of spectroscopy with high separating ability of chromatography, have become the most effective measurements of mercury currently [23]. Generally, instrumental analysis of mercury achieves high sensitivity and good accuracy but low detection level of below ppm. Consequently, it is only suitable for the measurement of trace level mercury to some extent [10, 16, 22, 24]. For the solutions at a middle or high mercuric concentration, superdilution is an indispensable procedure before the instrument analysis. In addition, the instrumental methods are laborious and expensive and then could not be widely applied to routine analysis. As compared with the instrumental techniques, the gravimetry and volumetry have simple experimental operation and measurement facility and are particularly suitable for the direct analysis of mercury-ion solution at middle and even high concentrations [24]. Furthermore, the commonly used volumetries of mercury, which include *Volhard* [25], *EDTA* [26], and dithizone

Correspondence: Xin-Gui Li, Key Laboratory of Advanced Civil Engineering Materials, Institute of Materials Chemistry, College of Materials Science and Engineering, Tongji University, 1239 Siping Road, Shanghai 200092, China. E-mail: adamxgli@yahoo.com

methods, are more applicable for the macro analysis of mercury ions. Although some primary gravimetries, such as electrolytic, cinnabar, mercurous chloride, periodate mercury, and *Reineckate* methods [24], have been reported for mercury determination, their sensitivity and accuracy are not as high as that of volumetries.

Since the dithizone method ordinarily uses toxic tetrachloromethane, another two volumetries are more commonly used in titration of mercury(II) ion recently. The *EDTA* method could be performed in direct titration or back titration, but the *Volhard* method is a direct titration [27, 28]. In fact, the *EDTA* and *Volhard* volumetries play important roles in macro analysis of mercury(II) ion. Analogically the macro analysis of mercury(I) ion is also based upon these two methods, just adding an oxidation of mercury(I) ion by strong oxidant-like nitric acid and potassium permanganate before titration. Therefore, the two-titration techniques have been currently developed to analyze mercury(I) ion samples. Note that incorporating nitric acid-hydrochloric acid and then heating are a rigorous experimental condition, because this procedure will result in evaporation of toxic elemental Hg (Hg°). The presence of potassium permanganate will result in a red solution that interferes with the estimation of titration end-point. Consequently, excessive oxalic acid should be added to decolorize the color of the residual potassium permanganate. Unfortunately, the reduced product of oxalic acid, $CO_3{}^{2-}$, will react with mercury(II) ion and form the precipitation of milky $HgCO_3$, especially in high-concentration mercurous solution. Apparently, oxalic acid should be replaced by other suitable reducer. $FeSO_4$ has been chosen in this study because its oxidized product Fe^{3+} is exactly the indicator of the following *Volhard* method. Considering that the complexation constant of the Fe-*EDTA* complex is very large, thus the *EDTA* could not be used regardless of a high complexation constant of the Hg-*EDTA* complex. Therefore, a method for the simple titration of $Hg^{2+} - Hg_2^{2+}$ mixed solution using NaSCN as titrant would be necessary. However, this special titrimetry which could be facilely utilized to determine the Hg^{2+}– Hg_2^{2+} mixed solution (10 mg/dm^3–10 g/dm^3) has not been found till now to the best of our knowledge. The novelty of this study lies in the proposition of a better suited reducing agent that would allow the use of the *Volhard* titration even after oxidation of Hg(I) to Hg(II) by $KMnO_4$.

Results and discussion

Determination of known Hg_2^{2+} and Hg^{2+} concentrations

Ten parallel experiments are applied to titrate mercury-ion solutions at a known concentration of Hg_2^{2+} and Hg^{2+} ions under the same condition as listed in Table 1. The average concentration of the mercury solution $\overline{C}$ is calculated to be 0.967 m*M* with an average relative error $\overline{E}$ of 1.09%, standard deviation *S* of 0.0145 m*M*, and relative standard deviation (*RSD*) of 1.50% through the following Eqs. (1)–(5):

$$\overline{C} = (C_1 + C_2 + C_3 + \cdots + C_n)/n \quad (1)$$

$$E = [(A - B)/B] \times 100\% \quad (2)$$

$$\overline{E} = (|E_1| + |E_2| + |E_3| + \cdots + |E_n|)/n \quad (3)$$

$$S = \sqrt{\frac{\sum_{i=1}^{n}(C_i - \overline{C})^2}{(n-1)}} \quad (4)$$

$$CV = (S/\overline{C}) \times 100\% \quad (5)$$

where C_i denotes the concentration of the mercury solution measured in each experiment, *n* is the number of parallel experiments, *A* denotes observed value, and *B* denotes true value.

It is found from Table 1 that the total concentration of Hg_2^{2+} and Hg^{2+} mixed solution could be determined by this method with a very low relative error, which means that this method has higher detection accuracy than the traditional gravimetries and volumetries. Small *RSD* shows higher data stability of this method than a common digital ap-

Table 1 Titration data of the solution containing Hg_2^{2+} and Hg^{2+} ions (0.97 m*M*)

Serial No.	Mercuric-ion concentration/m*M*	Relative error *E*/%
1	0.98	+1.0
2	0.96	−1.0
3	0.99	+2.1
4	0.98	+1.0
5	0.96	−1.0
6	0.95	−2.1
7	0.94	−3.1
8	0.97	0
9	0.98	+1.0
10	0.97	0

paratus for the determination of mercury based on cold-vapor atomic absorption spectrometry within 5% [29]. Since the MnO_2 precipitates formed during the pretreatment have been reduced to soluble Mn^{2+}, an earlier appearance of the titration end caused by the precipitates would be avoided. By the way, it is very hard to give information on the ratio of Hg(I) and Hg(II). However, this method could simply be utilized to directly analyze the solution in any ratio of Hg(I) and Hg(II), including pure Hg(I) or Hg(II).

Determination of a known Hg^{2+} concentration

This method could be employed to titrate not only the sample containing both Hg_2^{2+} and Hg^{2+} ions, but also the sample containing only Hg^{2+} ion. A detailed operation approach is described as follows: a 10.0 dm^3 9.97 m*M* standard Hg^{2+} solution was added to a 50 cm^3 conical flask, the remainder experimental operations are just the same as the approach presented in Experimental Section. Finally, this solution was titrated with 8.65 m*M* standard NaSCN solution. Ten parallel experimental results based on this testing method are shown in Table 2. It is found that $\overline{C}$ is 10.04 m*M* with an $\overline{E}$ of 0.82%, *S* of 0.077 m*M*, and *RSD* of 0.77%. The analytical data by the traditional *Volhard* method are also listed in Table 2 for comparison. The $\overline{C}$ by the traditional *Volhard* method is 9.972 m*M* with an $\overline{E}$ of 0.68%, *S* of 0.082 m*M*, and *RSD* of 0.82%. Apparently, the results are similar, indicating that the titrimetry developed in this study is also suitable for the determination of mercuric ion concentration of pure Hg^{2+} solutions.

Table 2 Titration data of the solution containing Hg^{2+} ion (9.97 m*M*) only

Serial No.	This titrimetry		Traditional *Volhard* method	
	Mercuric ion concentration	Relative error	Mercuric ion concentration	Relative error
	m*M*	*E*%	m*M*	*E*%
1	10.05	+0.80	10.05	+0.80
2	9.99	+0.20	9.99	+0.20
3	10.08	+1.1	9.95	−0.20
4	10.05	+0.80	9.86	−1.1
5	9.95	−0.20	10.08	+1.1
6	10.16	+1.9	9.99	+0.20
7	9.99	+0.20	9.86	−1.1
8	10.05	+0.80	10.05	+0.80
9	10.16	+1.9	10.08	+1.1
10	9.95	−0.20	9.95	−0.20

Practical application of this titrimetry

In an Hg^{2+} ion solution after adsorption on three aromatic amine polymers shown in Fig. 1, some Hg^{2+} ions could be reduced to Hg_2^{2+} ions. Thus, the remainder solution containing Hg^{2+} and Hg_2^{2+} ions simultaneously was determined by the described method. The titration data with standard NaSCN solution are summarized in Table 3. It is seen that this testing method was applicable to analyze the mercuric ion concentration in a range from 0.052 to 6.242 m*M*.

It seems that there is no perfect macro-analytical technique that could directly be employed to the analysis of an $Hg^{2+} - Hg_2^{2+}$ mixed solution. Generally, the macro analysis of mercuric samples is beyond the reach of instrumental analysis. If any instrumental analysis could be applied to the determination of the total concentration of Hg_2^{2+} and Hg^{2+} ions in solutions, super dilution is prerequisite before the instrument analysis. Apparently, this titrimetry developed here is the best choice for these analyzes.

Characteristics of this titrimetry

The total concentration of Hg_2^{2+} and Hg^{2+} ions in acidic, neutral, and even basic media could be accurately determined using this titrimetry. This method

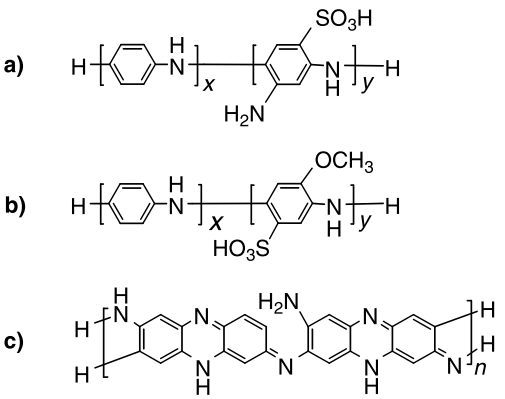

Fig. 1 The structures of three aromatic amine polymers as mercuric ion sorbents exhibiting a reduction of some Hg^{2+} ions to Hg_2^{2+} ions. a) Aniline/2-sulfo-1,5-phenylenediamine copolymer; b) aniline/5-sulfo-2-anisidine copolymer; c) *m*-phenylenediamine polymer

Table 3 Experimental results of the sample solutions containing different ratios of Hg_2^{2+} and Hg^{2+} ions by this titrimetry

Sample No.	Hg_2^{2+}:Hg^{2+} molar ratio	$KMnO_4$ solution consumed/drop	$FeSO_4$ solution consumed/drop	NaSCN solution consumed/cm^3	NaSCN conc./m*M*	Mercuric ion conc./m*M*
1	0:100	1	2	0.30	3.46	0.052
2	0:100	1	2	1.25	3.46	0.216
3	0:100	2	3	2.05	3.46	0.354
4	2:98	1	3	14.50	8.61	6.242
5	4:96	1	3	10.00	8.61	4.305
6	9:91	2	4	7.70	8.65	3.330
7	9:91	2	4	7.75	8.65	3.352
8	11:89	3	6	9.75	8.65	4.217
9	11:89	4	7	12.60	8.65	5.450
10	20:80	2	3	7.85	3.46	1.358
11	20:80	5	10	8.45	8.65	3.655
12	20:80	5	10	8.70	8.65	3.763
13	25:75	3	4	4.35	8.65	1.881
14	29:71	7	10	8.30	8.65	3.590
15	29:71	7	30	8.35	8.65	3.611
16	40:60	2	3	4.35	3.46	0.752

could be employed to analyze not only the sample containing both Hg_2^{2+} and Hg^{2+}, but also the solution of single Hg_2^{2+} or Hg^{2+}. This new method could own several advantages: simple operation, short analyzing time, good reproducibility, low detection limit of 50 μM (10 mg/dm^3), and wide concentration range from 200 μM to 50 m*M* (40 mg/dm^3–10 g/dm^3). The sudden advent of titration end-point is very sensitive in experiment and the concentration calculated from the equivalence point scarcely depends on titration rate. Moreover this technique could be a wet chemical analogue to instrumental approaches in use for a study on mercury ions in natural waters. Therefore if species of mercury ions in the titrand solution is not clear, this titrimetry is an advisable choice. In one word this technique has simpler operation, higher data stability, lower analytical cost, and easier popularization than traditional instrumental analysis.

In particular, note that during the traditional *Volhard* method, the addition of excessive oxalic acid would reduce potassium permanganate into precipitated MnO_2, accompanied with the formation of $HgCO_3$ and HgC_2O_4 precipitates. Additionally, the excess of oxalic acid will react with indicator Fe^{3+} ions added in a later step, leading to an increased dosage of indicator and then forming new precipitate. All these precipitates will disturb the observation of solution color. These negative effects will appear more serious in high concentration mercurous solution. Fortunately, the oxalic acid is completely replaced by $FeSO_4$ in this new titrimetry. Accompanying the oxidation of the mercury(I) to the mercury(II), red Fe(III) ion formed could be an indicator with a very sensitive color change at the end-point.

Important hint for successful titrimetry

Mercury thiocyanate, $Hg(SCN)_2$, is slightly soluble in water with a solubility of 70 mg/100 g (*i.e.*, 2.215 m*M*) at 25°C [30]. During titration once the $Hg(SCN)_2$ formed in titrand solution is more than its solubility, mercury thiocyanate will appear as precipitate in the solution. At that moment vigorously shaking the solution is indispensable, otherwise fresh $Hg(SCN)_2$ precipitate would adsorb Hg^{2+}, which may cause an earlier end-point. The vigorous shaking, especially for the concentrated mercury solution, also helps to release the adsorbed Hg^{2+} and then achieves a fast equilibrium. The last key drop of thiocyanate solution, giving a faint permanent red-brown solution, should be the real end-point [31].

For the analysis of wastewater samples containing mercury ions, the identification of mercury(I) ion is necessary. If there is no mercury(I) ion in the sample, the addition of $KMnO_4$ and $FeSO_4$ to the titrand solution is obviously redundant. A simple identifying method of mercury(I) ion is described as follows: 3 cm^3 0.2 m*M* NaCl solution were added in 3 cm^3 sample. If an ivory-white precipitate appears, and the ivory-white precipitate changes into black immediately when ammonia water is added, this phenom-

enon indicates the presence of mercury(I) ion, as depicted in Eqs. (6)–(7).

$$Hg_2^{2+} + 2Cl^- \rightarrow Hg_2Cl_2 \downarrow \quad (6)$$

$$Hg_2Cl_2 + 2NH_3 \rightarrow HgNH_2Cl \downarrow + Hg \downarrow + NH_4Cl \quad (7)$$

Conversely, no precipitate implicates the absence of mercury(I) ion in the solution. Nevertheless, trace mercury(I) ion (less than 2 ppm) is beyond this identification.

Meanwhile before the titration with standard NaSCN solution, some metal ions, such as copper and silver ions, as well as strong oxidants and nitrogen oxide, which could react with SCN^-, should be removed to avoid their interference reaction during the titration process [24].

In conclusion, a titration methodology, with simple operation, short processing time, and good reproducibility, has been established and successfully applied in the determination of the total concentration of Hg_2^{2+} and Hg^{2+} ions. Not only the sample containing both Hg_2^{2+} and Hg^{2+} ions, but also the solution containing either Hg_2^{2+} or Hg^{2+} ions could be titrated by this method. Besides, this improved titrimetry possesses low detection limit, wide analytical concentration range, and a small relative error. So this new method presented here will demonstrate useful in fields such as the determination of mercury-ion concentration in pesticides, medicine, and metallurgy, and measurement of total concentration of mercury at a high mercuric concentration.

Experimental

Caution: Mercury compounds and concentrated acids should be handled with gloves in a fume hood. Mercuric solutions are kept in the dark and near ambient temperature until processed.

Chemicals

The sodium thiocyanate was purchased from Linghu Fine Chemical Co (Huzhou, Zhejiang) and disodium ethylenediaminetetraacetic acid (*EDTA*), silver nitrate, and nitric acid were purchased from China Medicine (Group) Shanghai Chemical Reagent Co. The mercuric nitrate, ammonium iron(III) sulfate, potassium permanganate, ferrous sulfate, and sodium chloride were purchased from Shanghai General Chemical Reagent Works. All these chemical reagents were analytical reagent grade and used without further purification.

Preparation and standardization of the solutions

Solutions of 390 m*M* $(NH_4)Fe(SO_4)_2$, 50 m*M* $KMnO_4$ and 100 m*M* $FeSO_4$were all made in freshly boiled, distilled, deionized water as indicator, oxidant, and reductant [25]. HNO_3 solution (3 *M*) was also made in freshly boiled, distilled, deionized water using a 65–68% *w*/*v* concentrated nitric acid (14.44–15.11 *M*) [25] and any nitric acid used in titration must be free from HNO_2, which forms a complex with SCN^- ions. Stock solution of sodium thiocyanate (NaSCN 100 m*M*) was prepared in distilled, deionized water and standardized with a standard silver nitrate solution, with ammonium iron(III) sulfate as indicator. The stock solution of mercuric nitrate ($Hg(NO_3)_2$100 m*M*) was prepared in distilled, deionized water and standardized by titration with a standard *EDTA* solution with xylenol orange as indicator. The NaSCN and $Hg(NO_3)_2$ standards were freshly prepared on each occasion by serial dilution of the standard stock solutions without additional acid. The standard *EDTA* solution was prepared in distilled, deionized water with solute *EDTA* weighted accurately.

Background and theory

Complexometric titration in a dilute nitric acid medium (0.1–1.0 *M*) is employed in this titrimetry of mercury ions. Before the titration course of mercury ions, Hg_2^{2+} ions must be oxidated by an excess of $KMnO_4$ solution firstly. Then the residual $KMnO_4$ was reduced by the treatment with $FeSO_4$, meanwhile the Fe^{3+} ions produced in this reaction could be a part of indicator in the later fitting approach, after which the solution is ready for titration with standard NaSCN solution as titrant and ammonium iron(III) sulfate as indicator. The principle of this method is described below:

1) The oxidation of mercury(I) ion

MnO_4^- ion as oxidant was reduced by mercury(I) ion forming MnO_2 as russet floccule in weak acid medium:

$$2MnO_4^- + 3Hg_2^{2+} + 8H^+ \rightarrow 2MnO_2 \downarrow + 6Hg^{2+} + 4H_2O \quad (8)$$

The excess of MnO_4^- ion was also reduced by Fe^{2+} ion added later:

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O \quad (9)$$

After the snuff color in the solution disappeared, more $FeSO_4$ solution was added to reduce the russet precipitated MnO_2:

$$MnO_2 + 2Fe^{2+} + 4H^+ \rightarrow Mn^{2+} + 2Fe^{3+} + 2H_2O \quad (10)$$

2) Quantitative complexation of mercury(II) ion and thiocyanate ion

As the 1st-order complex $Hg(SCN)^+$ between Hg(II) and thiocyanate ion with molar ratio of 1/1 hardly ever exists, the complexation mainly follows the reaction equations:

$$Hg^{2+} + 2SCN^- \rightarrow Hg(SCN)_2 \quad (11)$$

$$Hg(SCN)_2 + SCN^- \rightarrow Hg(SCN)_3^- \quad (12)$$

$$Hg(SCN)_3^- + SCN^- \rightarrow Hg(SCN)_4^{2-} \quad (13)$$

The stepwise formation constants, K_2, K_3, and K_4, of these three complexation reactions are 1.41×10^{17}, 513 and 52.5 [32, 33]. It is seen that K_2 is much larger than K_3 and K_4,

suggesting that the latter two complexations could be ignored. Consequently among the three complexes between mercury(II) and thiocyanate ions, the fraction of $Hg(SCN)_2$ is almost 100%. In other words the vast majority of mercury(II) ion in the solution exists as mercury(II)/thiocyanate (1/2) complex. Therefore mercury(II) ion could be satisfactorily titrated with thiocyanate solution, assuming that at its equivalence-point, the mercury(II)/thiocyanate complex (1/2), $Hg(SCN)_2$, is the only speciation for mercury(II) in the sample. In the titration course, colorless complex $Hg(SCN)_2$ was formed firstly [34]. On approaching its equivalence-point, the quantity of Hg^{2+} ions reduces sharply by adding SCN^- ion solution [35, 36]. Once slightly excessive thiocyanate ion was added, a red thiocyanate-iron(III) complex appeared in the titration solution, indicating the end of the titration. It should be noticed that Fe(III)-SCN^- was very frequently used as typical indicator for the *Volhard* method because of its suitable K_1 of 199.53. If the K_1 were significantly higher, Fe(III) would compete with mercury ions for the complexation with SCN^-, destroying quantitative relationship between titrant (SCN^-) and titrand (mercury ion).

Determination procedure

The detailed titration method is as follows: a 10.0 cm^3 mercury-ion solution was added to a 50 cm^3 conical flask, after which an excess of 50 m*M* $KMnO_4$ was dropped to oxidize possible mercury(I) into mercury(II). Then the residual $KMnO_4$ was reduced by dropwise adding 100 m*M* $FeSO_4$. Subsequently 3 cm^3 390 m*M* $(NH_4)Fe(SO_4)_2$ and 5 cm^3 3 *M* HNO_3 solutions were mixed with the mercury(II) solution. Since then the solution is ready for titration with standard NaSCN solution and this titration system has a very sensitive color change at the end-point. Therefore the total concentration of mercury ion in titrand solution could be calculated from the volume of standard sodium thiocyanate solution consumed in titration.

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